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A Comparison of the Toxicity and the Diffusion in a Column of Grain, of Chlorpicrin, Carbon Disulphide, and Carbon Tetrachloride

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A COMPARISON OF THE TOXICITY TO INSECTS AND THE DIFFUSION IN A COLUMN OF GRAIN, OF CHLORPICRIN, CARBON DISULPHIDE, AND CARBON TETRACHLORIDE

By A. L. Strand

INTRODUCTION

For fifty or more years carbon disulphide has been used as a fumigant against a wide variety of insect pests. The early work was concerned mainly with showing its toxicity, but the more recent work has dealt with the effects of temperature, leakage, and other factors in connection with its use. Likewise, it has been well established that carbon tetrachloride, altho possessing the important advantage of being non-inflammable, is a much weaker fumigant than the disulphide. Within the last ten years the high toxicity of chlorpicrin to insects has been thoroly demonstrated. This fact and the knowledge that it is non-inflammable have led workers in many different countries to look upon chlorpicrin as one of the most promising substances in the field of insect fumigation. These three materials have been compared, but they have not been compared under controlled conditions over a range of temperatures.

Because of the irritating effects of chlorpicrin on man, and owing to certain of its physical properties, the practical use of chlorpicrin as a fumigant has centered on the technic for its application. As one of its chief uses is the treatment of infested grain, more accurate knowledge was desired concerning its diffusion in grain. It was thought that the manner of its diffusion would be the initial step in arriving at the most intelligent method of application. Furthermore, as chlorpicrin, when used as a fumigant on stored products, is necessarily compared with the better-known carbon disulphide and carbon tetrachloride, it was believed that the diffusion of these chemicals in grain should be studied at the same time. Consequently the purpose of this present work has been two-fold (1) to determine the relative toxicity of carbon tetrachloride, carbon disulphide, and chlorpicrin to insects at various temperatures; and (2) to study the manner of diffusion of these chemicals when applied to threshed grain in storage. The two divisions of the work will be taken up in that order.

Acknowledgment

The writer wishes to acknowledge the helpful suggestions of Dr. R. N. Chapman, as to both the initiation of the problem and it development.

TOXICITY TO INSECTS OF CHLORPICRIN, CARBON DISULPHIDE, AND CARBON TETRACHLORIDE

Historical Summary

Piutti and Bernardini (1917)[1] and Moore (1917) were the first to point out the high toxicity of chlorpicrin to insects. Moore rated it above carbon tetrachloride and carbon disulphide. In millionths of gram molecules necessary to kill house flies in 400 minutes, the three compared as follows: chlorpicrin, 1.7; carbon tetrachloride, 161.9, and carbon disulphide, 286.3. In a later publication, Moore (1918) stated that, molecule for molecule, chlorpicrin was 283 times as toxic as carbon disulphide. In the same paper he showed that chlorpicrin at the rate of half a pound to 1000 cubic feet killed the bean weevil (*Bruchus obtectus* Say), the Angoumois grain moth (*Sitotroga cerealella* Oliv.), the Indian meal moth (*Plodia interpunctella* Hbn.), and the Mediterranean flour moth (*Ephestia kuehniella* Zell.), but did not kill the confused flour beetle (*Tribolium confusum* Duv.) deeper than an inch in the flour. For the last named insect, he stated that a concentration of 1 or 2 pounds of chlorpicrin should be used, and that to obtain similar results with carbon disulphide from 3 to 5 pounds per 1000 cubic feet at temperatures above 65° F. would be required. Moore (1918) also pointed out that chlorpicrin gave good results at temperatures below 60° F.

Bertrand, Brocq-Rousseu, and Dassonville (1919c) found that they could kill *Tribolium navale* Hbst. in 1 hour and 20 minutes at a concentration of 30 grams per cubic meter (slightly less than 2 pounds to 1000 cubic feet) at a temperature between 14° and 19° C., and that at reduced concentrations longer periods were required. They demonstrated that *T. navale* was much harder to kill than the grain weevil (*Sitophilus*) and that a separation of the two insects in a mixed infestation could be effected with chlorpicrin. These authors in another paper (1919b) have given the best account of the effect of temperature on the insecticidal power of chlorpicrin. Data given show that at 37° C. 20 grams per cubic meter killed *Sitophilus oryza* L. in 31 minutes while at 0° C. 4 hours was required.

Wille (1921a) has reviewed the work of Bertrand and his associates and in another paper (1921b) states that chlorpicrin at the rate of 30 cc. per cubic meter kills the grain weevil in 6 hours.

Bertrand and Rosenblatt (1919) showed that, out of eight chemicals used, chlorpicrin was most toxic to *Bombyx neustria* L., even more so than hydrocyanic acid gas. The insects were subjected to measured amounts of air and poisonous vapors for periods from 10 minutes to

[1] The writer has not seen this reference.

1 hour. The toxicities were ranked as follows: chlorpicrin, hydrocyanic acid, chloracteone, benzyl bromide, carbon disulphide, chloroform, carbon tetrachloride, and ether. No difference was noted in the relative toxicity of these substances toward different species of insects. Again Bertrand, Brocq-Rousseu, and Dassonville (1919a) found that the bedbug (*Cimex lectularius* L.) was killed at a concentration of 20 grams of chlorpicrin to 1 cubic meter in 33 minutes (or a minimum of 20 minutes), and that at concentrations of 1 gram per cubic meter 12 hours and 22 minutes were required. The time required to kill with intermediate amounts was proportional to the dosage.

Burchardt (1920) believed that chlorpicrin was not so good a fumigant as carbon disulphide. In fumigating sacks of grain in a granary he used 10 grams of chlorpicrin per cubic meter and failed to secure a kill in 48 hours, while with carbon disulphide good results were secured in from 6 to 24 hours. The insects concerned were *Sitophilus, Laemophloeus ferrugineus* Steph., and *Orysaephilus surinamensis* L.

Feytaud (1920) states that at 15° C. 20 milligrams of chlorpicrin per liter caused the death of all termites (*Leucotermes lucifugus* Rossi) in 2 hours. Smaller concentrations required longer time. Four and one-half milligrams per liter killed the insects or poisoned them beyond recovery in 24 hours. At 20° C., 2 milligrams per liter killed the insects in 12 hours.

Neifert and Garrison (1920) compared chlorpicrin with hydrocyanic acid and, in general, for the fumigation of stored products found it more poisonous to insects than the cyanide.

Chapman and Johnson (1925) have corroborated and extended some of the experiments of Bertrand, Brocq-Rousseu, and Dassonville, and found as well that the time to secure a 100 per cent kill and the concentration of the chlorpicrin bear an inverse ratio to each other. They state that "considering time, temperature, and concentration, a low value for any one could be compensated for by a correspondingly high value to one or more of the others." No relationship was found between relative humidity and toxicity. At low concentrations chlorpicrin showed variations in the time necessary to secure 100 per cent kills, but at high concentrations it was very definite and exact in that respect. It was pointed out that the susceptibility of different species of insects is different, and that the confused flour beetle (*Tribolium confusum* Duv.) is more resistant than the grain weevils and requires from 30 to 50 per cent more chlorpicrin to kill it.

Altho the number of references to carbon disulphide in the literature is very large, there is little definite information on the minimum time required to kill at different temperatures or at different concentrations.

Hinds (1909) called attention to the importance of temperature in carbon disulphide fumigations. In this article he gives the minimum time to kill 100 per cent of *Bruchus chinensis* L. as 43 minutes, presumably at a temperature of 68° F. in saturated air.

Hinds and Turner (1910) again pointed out the importance of temperature in fumigating with carbon disulphide. They showed that even high concentrations at low temperatures failed to kill *Sitophilus oryza* L. and stated that "it requires but a few hours to kill the weevils if a strength of gas equal to one-quarter of a saturated atmosphere can be maintained and providing the temperature is high enough to insure a considerable degree of vitality on the part of the insects."

Chittenden (1911, pp. 37-38 and 42-46) showed that poor results with carbon disulphide were secured at temperatures below 50° F. even when a large amount of the fumigant was used.

Portchinsky (1913) found that at 14°-16° R. (64°-68° F.) in a tight place, it was necessary to apply not less than 7 pounds of carbon disulphide per 1000 cubic feet to kill stored products insects in 48 hours. To kill in 24 hours, 10.5 pounds was necessary.

Girault (1912a, p. 77) states that "our recent experiments carried on with great care and duplicated many times have convinced us that an effective fumigation requires 10 pounds of carbon disulphide to every 1000 cubic feet of space to be treated. At this strength we have found the fumigant effective against all granary pests and at all temperatures."

Larson (1924) found that large quantities of beans required a longer exposure or a greater quantity of the fumigant, but that small quantities could be successfully rid of weevils with 3 pounds of carbon disulphide per 1000 cubic feet of space at 58° F. in 24 hours, or with 1.5 pounds in 48 hours.

Neifert and associates (1925) found carbon disulphide far more toxic than carbon tetrachloride. In one instance carbon tetrachloride at the rate of 8.58 pounds per 1000 cubic feet at 30° C. failed to kill any *Sitophilus oryza* L. or *S. granarius* L. in 24 hours. However, a concentration of 29.41 pounds at 23.5° C. killed 100 per cent of the *S. oryza, S. granarius,* and *Tribolium confusum* Duv. present. On the other hand, carbon disulphide at the rate of 2.33 pounds per 1000 cubic feet at 27.5° C. killed 100 per cent of *S. oryza, S. granarius,* and *Plodia interpunctella* Hbn. in 24 hours. These particular experiments were carried out in glass flasks.

Back and Cotton (1925) have compared a mixture of ethyl acetate and carbon tetrachloride with carbon disulphide but did not determine the minimum time to kill with either substance.

Evidently Britton (1908, p. 275) was the first to substitute carbon tetrachloride for carbon disulphide. He reported experiments with tetrachloride when used as a fumigant against scale insects.

Morse (1910) was the first to report the results of using carbon tetrachloride instead of carbon disulphide against museum pests. He found that where 1 pint of the disulphide was sufficient for 50 cubic feet of space, 1 quart of the tetrachloride was required in order to secure the same killing strength.

Shafer (1915, pp. 61-63) reports on some experiments carried on with carbon tetrachloride in 1910 at the Michigan Agricultural College. A series of comparative tests showed that six times as much tetrachloride as disulphide was necessary to accomplish the same results at about 70° F.

Chittenden and Popenoe (1911), with 1.5 pounds of carbon tetrachloride per 1000 cubic feet, did not secure a kill of *Orysaephilus surinamensis* L. or *Laemophloeus minutus* Oliv. in 48 hours, or with twice that amount. With 6 pounds per cubic feet the larvae of *Tenebroides* were unharmed, less than half of the larvae of *Ephestia kuehniella* Zell. were killed, and the common weevils found in grain were living, altho they seemed to be paralyzed. The period of exposure in this case was 24.5 hours. At 10 pounds per 1000 cubic feet, bean weevils (*Bruchus quadrimaculatus* Fab.) were killed in 24 hours, but Cadelle larvae (*Tenebroides mauritanicus* L.) moved slowly after the fumigation. The temperatures during these experiments were not given.

Numerous other authors have made the general statement that carbon tetrachloride can be used as a substitute for carbon disulphide, but all agree that it must be applied in much greater amounts.

Experiments Conducted as a Means of Determining the Relative Toxicity of Carbon tetrachloride, Carbon disulphide, and Chlorpicrin at Different Temperatures

The experiments were performed within a constant temperature and humidity cabinet. The insects used were adult confused flour beetles (*Tribolium confusum* Duv.) taken from one infestation, feeding on cornmeal. The reasons for selecting this species were its availability and the fact that other investigators had found *Tribolium* to be more resistant to the action of chlorpicrin than many other insects (Moore, 1918; Bertrand, Brocq-Rousseu, and Dassonville, 1919a). Altho the species is undoubtedly also resistant toward the action of carbon disulphide and carbon tetrachloride, it was thought to be as good a basis as any on which to compare toxicity.

Method

The apparatus within which the insects were actually subjected to the action of the fumigants and which was used within the constant temperature and humidity machine is shown in Figure 1. This consisted, essentially, of a large bell jar and a ground-glass plate on which the bell jar rested.

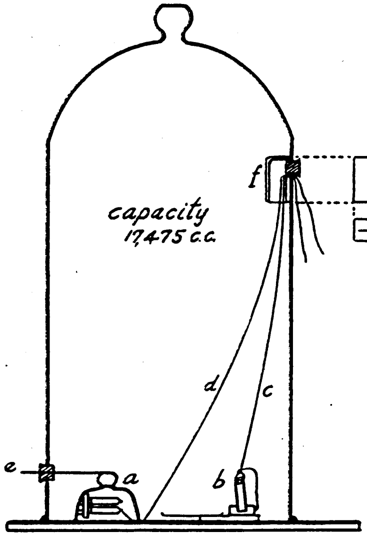

Fig. 1. Diagram of Fumigation Chamber

This chamber was so designed as to shield the insects during the evaporation of the liquid fumigant and to permit the withdrawal of individual cages after various exposures.

Five adults of the confused flour beetle were placed in each of from 10 to 14 cylindrical cages, 1.5 inches long and 0.5 inch in diameter, which were made of rather coarse bolting cloth. These cages were all connected at intervals of 35 inches to a linen thread (d) in order that they could be withdrawn from the bell jar after various lengths of time. The cages containing the insects were implaced on a rack within a very small bell jar shown at "a", which at the beginning of the experiment was sealed to the ground-glass plate with plasticine. From the knob on this small bell jar a cord (e) extended through a rubber stopper which was inserted in a hole through the fumigation jar. The insects were put in the small ball jar to protect them from the action of the gas until the gas had reached its full strength by the complete evaporation of the liquid, in which form it was applied.

The liquid fumigant was measured with a 1-cc. pipette having a glass stop-cock at the lower end, and graduated to 0.01 cc.[2] The ma-

[2] The smallest amount of any material measured in this set of experiments was 0.16 cc. or, in later experiments with the same pipette, 0.08 cc.

terial was measured directly into a small glass vial (b) to which was attached a thread (c) running to the outside of the large jar. By means of this thread the vial could be uncorked and its contents dumped on crumpled filter paper.

The threads (c and d) ran first through a slit (g) in the rubber dam with which the lower end of the trap (f) was covered and thence through a hole in the large jar which was fitted with a rubber stopper. This part of the apparatus was designed to prevent, as far as possible, loss of fumigant when a cage containing insects was withdrawn.

After the small bell jar was in place and the fumigant was in the vial and tightly stoppered, the entire apparatus was placed in the temperature cabinet. The large jar was then raised about ten inches from the ground-glass plate and left in that position long enough for the apparatus to come to the temperature of the cabinet and the air within the fumigation jar to assume the same relative humidity as the air in the cabinet. Then the large jar was lowered on the base plate and sealed around the edges with plasticine. The thread (c) which uncorked the vial and applied the liquid, was next pulled. When the last trace of liquid had evaporated, and after a few minutes additional time had been allowed, the cord (e) was pulled and the actual fumigation of the insects began. By means of that cord the small jar was not only pulled loose from the base plate but was also made to stand up on its side. In this way the cages of insects on the rack within the jar were lifted well off the floor of the fumigatorium.

Near the attachment of the first cage of insects to the thread (d) was also attached a small glass tube closed at the ends with bolting cloth and containing 5 adult beetles. Immediately after the small bell jar was raised, this tube was drawn to such a position that the insects within it could be easily observed by the operator standing outside the glass door of the temperature cabinet. With chlorpicrin, this observation vial was of considerable aid in estimating the time to begin the periodic withdrawal of the cages. But with carbon disulphide and, particularly, with carbon tetrachloride, the time necessary to cause the insects in the observation vial to become inactive seemed to bear little or no relation to the time necessary to kill them.

After each vial was withdrawn it was left beside the fumigation jar until the completion of the experiment. Then the insects were removed from the cages[3] and placed in short glass vials which were kept at room temperature (22° C.) for 24 hours. At the end of that period the percentage of insects killed was noted.

[3] On account of the adsorption of the gases, especially chlorpicrin, on the cages, it was necessary to heat them for a time after each experiment in order to make sure that when they were again sealed in the small jar enough gas would not be released to cause an error in the experiments. For the same reason all parts of the apparatus where any adsorption could take place were heavily coated with paraffin.

Materials Used

The chlorpicrin used was furnished by a chemical company of New York State. The carbon disulphide and carbon tetrachloride were technical grades obtained from the chemical storehouse, University of Minnesota.

Manner of Checking Temperature and Percentage of Relative Humidity

As the air in the type of constant temperature and humidity machine used is continually in motion at the rate of about 15 feet per second, a very even temperature is maintained through the cabinet. The recording thermometer (dry bulb) was checked with a standard centigrade thermometer suspended within the cabinet. The percentage of relative humidity, as shown by the readings of the recording wet and dry bulb thermometers, was checked by a set of standard wet and dry bulb thermometers situated in the air stream near the top of the cabinet.

In a few cases the wet bulb temperature showed considerable variation, indicating changes in relative humidity; but in all such cases, previous to and at the time of lowering the large bell jar to the base plate, a relative humidity of 50 per cent obtained. After the jar was sealed and as long as a constant temperature was maintained, the relative humidity within the fumigation jar remained at 50 per cent. On all the charts, small variations in the wet bulb temperature were due to having the door of the cabinet open long enough to withdraw one of the cages.

Details of Experiments on Toxicity

The first experiments were conducted at 30° C. and several concentrations of each fumigant were tried in order to select concentrations for all tests which would not require too greatly extended periods but which would fall, nevertheless, within the limits of concentrations commonly recommended for these fumigants. Perhaps 15 pounds per 1000 cubic feet for carbon disulphide was a little high. However, it was not expected that the toxicity of this material would be as well maintained as it was in the lower temperatures or 10 pounds per 1000 cubic feet would have been used. Concentrations greater than 15 pounds per 1000 cubic feet are often recommended for carbon disulphide. (See tables in Appendix, pages 32 to 44.)

Summary of Results of Toxicity Experiments on Chlorpicrin, Carbon Disulphide, and Carbon Tetrachloride

The results of the experiments on toxicity are summarized in Table I.

The curves shown on Figure 2 were plotted from the data of Table I.

TABLE I

MINIMUM TIME REQUIRED TO SECURE 100 PER CENT KILL OF *Tribolium confusum* DUV.
(Concentrations in pounds per 1000 cubic feet)

Temp. °C.	Chlorpicrin 1 lb.		Chlorpicrin 2 lb.		Chlorpicrin 3 lb.		Carbon disulphide 15 lb.		Carbon tetrachloride 20 lb.	
	hr.	min.	hr.	min.	hr.	min.	hr.	min.	hr.	min.
35	1	15	..	31	..	15*	..	..	..	..
						21	..	30	1	30
30	1	45	..	35	..	25	..	45	3	15
25	2	15	..	50	..	30	1	00*	7	00
							1	30	..	..
20	2	45	1	10	..	40	2	00	12	00?
15	3	50	2	00	1	00	3	00	..	..
10	4	30	2	40	1	20	3	30	..	..

* In cases where 100 per cent kills were followed by percentages less than 100, both times when the first complete kills occurred were recorded.

A comparison of the three fumigants at different concentrations at a temperature of 30° C. is given in Table II.

TABLE II

MINIMUM TIME REQUIRED TO KILL 100 PER CENT OF *Tribolium confusum* DUV.

Concentration per 1000 cu. ft.	Chlorpicrin	Carbon disulphide	Carbon tetrachloride
lb.	hr. min.	hr. min.	hr. min.
1	1 45	....	....
2	35	....	....
	45	....	....
3	25	....	....
4	16	....	....
10	....	1 05	....
	....	1 30	....
15	....	45	....
20	....	27	3 15
30	....	....	2 00
	....	....	3 00

Discussion of the Results on Toxicity

From the graphical summary of the results of the toxicity experiments (Figs. 2 and 3), it will be seen that comparisons of these fumigants must be made, not on concentration and time to kill alone, but also on temperature. For instance, at temperatures of 30° and 35° C. carbon tetrachloride shows a fair toxicity. However, considering the way the time to kill with this material decreases toward 25° and 20° C., it is not surprising that very indifferent results have been obtained with it in fumigating at normal temperatures (60° to 70° F.).

On the other hand, attention should be called to the continued high toxicity of carbon disulphide toward the lower temperatures. It was expected that close to 15° C. the time to kill with this fumigant would be greatly increased, but this was not the case. At 10° the insects were killed in 3 hours and 30 minutes with a concentration of 15 pounds per 1000 cubic feet of space. For this reason it seems improbable that many poor results of grain fumigations carried out at high concentrations but at fairly low temperatures (50° to 60° F.) can be explained solely from the standpoint that at such temperatures carbon disulphide has too low a toxicity, or that the insects are too inactive to be readily affected by the chemical.

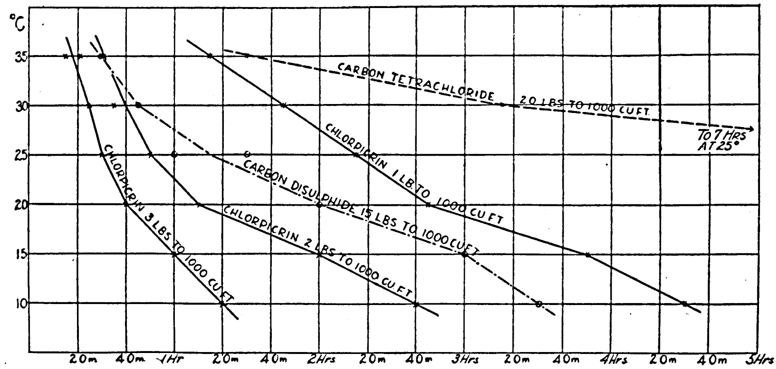

Fig. 2. Time and Temperature Curves for the Toxicity of Chlorpicrin, Carbon Disulphide, and Carbon Tetrachloride to *Tribolium confusum* Duv. at the Concentrations Indicated

The time and temperature curves for chlorpicrin (Fig. 3) are not straight line functions such as Bertrand, Brocq-Rousseu, and Dassonville (1919c) show in the case of chlorpicrin at a concentration of 20 grams per cubic meter (about 1.25 pounds per 1000 cubic feet). The

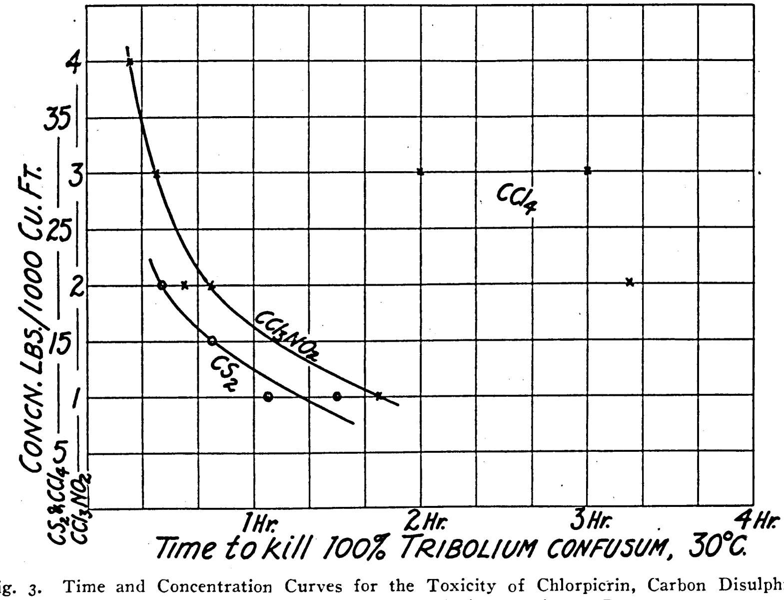

Fig. 3. Time and Concentration Curves for the Toxicity of Chlorpicrin, Carbon Disulphide, and Carbon Tetrachloride to *Tribolium confusum* Duv.

rice weevil, *Sitophilus oryza* L., was used by them to test the toxicity, and the difference between that insect and *Tribolium confusum* Duv. may possibly account for the discrepancy. Our general results on toxicity are in agreement with those of Bertrand and Rosenblatt (1919).

We do not believe that the statement of Moore (1918)—that molecule for molecule chlorpicrin is 283 times as toxic as carbon disulphide—is true or that chlorpicrin is 168.4 times as toxic, altho shown definitely enough by his work (Moore, 1917), conveys an entirely correct idea as to the relative toxicities of these two fumigants. On the contrary, we would say that at 20° C. 3 pounds of chlorpicrin per 1000 cubic feet is three times as toxic to insects as 15 pounds of carbon disulphide in the same space at the same temperature. In other words, chlorpicrin at 3 pounds per 1000 cubic feet at 20° C. is about 15 times as toxic as carbon disulphide, pound for pound. But 2 pounds of chlorpicrin at the same temperature is less than 15 times as toxic as a similar amount of carbon disulphide, and with 1 pound of chlorpicrin the ratio is still more reduced. Therefore, it seems unwise to make any definite statement regarding the relative toxicities without limiting it by the factors of temperature and concentration.

Throughout all the experiments a very marked difference was observed between the reaction of the insects to chlorpicrin and to either carbon tetrachloride or carbon disulphide. Even at a temperature of 10° C., which is but 2 or 3 degrees above the minimum effective temperature for *Tribolium confusum,* the insects in the presence of chlorpicrin became very active before death. On the other hand, carbon tetrachloride and carbon disulphide, even at low concentrations, seemed to have an anesthetic effect upon them. In some of the experiments with the tetrachloride, the beetles were inactive for several hours, but upon being taken out of the fumigant and aired they regained their normal activity in a very short time.

DIFFUSION OF CHLORPICRIN, CARBON DISULPHIDE, AND CARBON TETRACHLORIDE IN GRAIN

Historical Summary

Piutti and Mango (1920) state that the action of chlorpicrin when used on cereal at the rate of 20 cc. per cubic meter should be allowed to continue for 8 days where large quantities are to be disinfected. Piutti (1921) says that 20 cc. per cubic meter kills various insects when allowed to act for one week at temperatures from 59° to 68° F., whether the bin is empty or filled with grain. Yamamoto (1922) has said that the active period for chlorpicrin is from 48 to 70 hours, when applied at the rate of 0.225 to 0.35 gram per cubic shaku, which is approximately a concentration of from one-half to three-quarters of a pound to 1000 cubic feet. Aside from these statements, from which one might infer that some such periods are required for the gas to diffuse through the grain, and Piedallu's (1923) remark about the detection of the odor of the gas at the bottom of a silo three weeks after the material was applied, we have seen no published work regarding the diffusion of this chemical in grain.

Dr. R. N. Chapman. of the Minnesota Agricultural Experiment Station, performed an experiment in July, 1922, which has a direct bearing on this problem. An elevator bin 63 feet high, containing 1000 cubic feet, filled with oats to a height of 60.5 feet, was fumigated with chlorpicrin. The chlorpicrin was diluted with carbon tetrachloride, 1 part chlorpicrin to 4 parts tetrachloride. Seven and one-half pounds of this mixture was applied at the top of the bin. Sacks made of bolting cloth and containing the grain weevil, *Sitophilus granarius* L., were placed at the surface of the grain and 12, 22, 32, 42, 52, and 60 feet below the surface, in order to determine the downward diffusion of the gas. After an exposure of 72 hours the results were as follows:

Distance from surface, ft.	0	12	22	32	42	52	60
Per cent insects dead	100	100	0	50	0	0	3

Moore (1918) says that "one of the chief advantages of carbon disulphide in the fumigation of grain is that its vapor is 2.5 times heavier than air and is thus able to sink down through a large mass of grain. Chlorpicrin vapor is about twice as heavy as that of carbon disulphide." He infers somewhat that the vapor of chlorpicrin should diffuse downward through grain more rapidly than the vapor of carbon disulphide.

General statements regarding the diffusion of carbon disulphide in grain are numerous in the literature. Almost every one who has recommended the use of carbon disulphide as a control measure for insects attacking stored products has stated that the vapors are approximately 2.5 times heavier than air and consequently sink downward through the grain. (Howard, 1893, p. 327; Smith 1896, p. 326; 1900, p. 8; 1903, p. 24; 1908, p. 34; Chittenden, 1897, pp. 22-23; Back, 1919, p. 26; 1922, p. 27; and many others.)

Girault (1912b, p. 87) states that "the vapor of carbon disulphide is a little over 2.5 times heavier than air, a point to be remembered in application, since it goes first to the bottom of the inclosure."

Hinds (1917, pp. 8-9) says "when carbon disulphide is applied to a bin of grain or similar material it has been found that the killing proceeds outward and downward from the point of application of the liquid and forms what may be called a 'cone of killing.' The apex of the cone is close to the point of application, and the base is against the floor or ground below." In the same publication he also states that "in large masses of corn, containing more than 1000 bushels, where the corn still has the husk on, it is possible to kill enough insects in the interior of the mass to pay for the fumigation, even if the mass is entirely open to the air above and around it. The husks in so large a mass serve to retain the gas long enough to kill weevil, etc."

Dean (1913, pp. 196-197) and King (1920, pp. 12-13), altho stating that the gas is heavier than air and sinks to the lower parts or bottom, are among those who felt the need of getting the carbon disulphide below the surface of the grain.

Fleming (1923) actually measured the diffusion of carbon disulphide in potting soil and, in general, found that the concentration of the gas varied directly with the depth from the surface of the soil and inversely with the distance laterally from the injection hole. He pointed out, however, that his method of measuring diffusion was open to serious objection in that the manner of withdrawing the samples to be analyzed tended to disturb the natural diffusion of the gas in the soil.

Neifert and associates (1925, p. 24) state that "the insecticidal action of a gas is greatly lessened by the presence of grain, probably because the grain absorbs many vapors in large quantities and because the grain mechanically interferes with the diffusion of the gas throughout the receptacle."

Method Used for Estimating Diffusion of Chlorpicrin, Carbon Disulphide, and Carbon Tetrachloride

Ordinary methods of gas analysis, such as used by Neifert and Garrison (1920, p. 10) for chlorpicrin or by Fleming (1923, pp. 22-24) for carbon disulphide, did not seem practical in working on the diffusion of gases in grain, chiefly because the samples of gas would have to be withdrawn and thereby natural diffusion would be altered.

During the world war active charcoal, which has the power of adsorbing almost any gas at an astonishing rate, was developed for use in gas masks. Since the war such active charcoals, manufactured as more or less standard products, have come into wide use in industry. Because of the great adsorption capacity of this material, it was readily determined that small amounts of it could be used to estimate fairly accurately the concentration of chlorpicrin, carbon disulphide, or carbon tetrachloride in any container. Consequently it was adopted as the best means at our disposal for working on the diffusion of gases in grain.

Tests of Known Concentrations of Gases Under Various Constant Temperatures with Active Charcoal

Method.—The active charcoal was held in glass tubes which were 2 cm. in inside diameter and 6.5 cm. long. This size was selected so that it could be used later in a grain sampler. Approximately 10 cc. of charcoal was used in one tube. After filling, the tube was closed tightly with a rubber stopper and weighed accurately to 0.0001 gram. A weight close to 5 grams was obtained by adding or taking out the small amount usually necessary. The stopper was removed just before the charcoal was exposed to a gas and replaced immediately after. After the exposure the tube was re-weighed and the adsorption in milligrams on 5 grams of charcoal in one hour was determined.

The supply of active charcoal was kept in a dessicator jar tightly sealed with vaseline. None of the charcoal was used more than once. The charcoal was not dessicated.

In making the tests, wide-mouthed bottles, with a capacity of 8650 cc. were used (Fig. 4). A tube of charcoal was placed in the container (a), which consisted of a large glass tube held firmly to the bottom of the jar with surgeon's tape. About the tube of charcoal a slightly larger tube was kept in order to prevent contamination from handling. As soon as the tube of charcoal with its protecting tube was placed in the container (a), the latter was tightly closed with a rubber stopper. To make the stopper especially tight, a small amount of vaseline was applied to it. From the stopper a cord extended through the cork used later to close the bottle at the top.

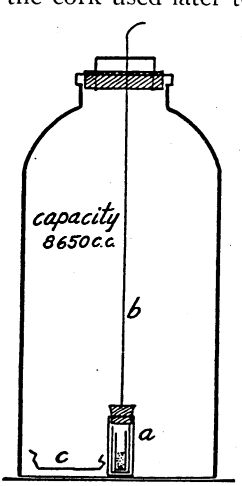

Fig. 4. Diagram of Apparatus in Which Tubes of Charcoal Were Exposed to Known Concentrations of Chlorpicrin, Carbon Disulphide, and Carbon Tetrachloride Under Constant Temperature and Humidity Conditions

A set of three of these bottles was placed in the temperature cabinet. The large corks at this stage of the operation were hung directly over the bottles by means of the wire loops which may be seen in the diagram. The cord (b) running through each cork was long enough that the stopper in the container (a) was not disturbed by the suspension of the cork several inches above the bottle. Time was allowed for the apparatus to assume the temperature and humidity conditions of the cabinet. In order to know when this stage had been reached, a thermometer was fastened inside of one of the bottles in such a position that it could be read through the glass door of the temperature cabinet. The closely fitting heavily paraffined corks were than put rather lightly in place.[4] Following this, the cork to each bottle was raised just enough to drop a small vial containing the correct amount of the fumigant on the filter paper at the bottom of the bottle. Then the corks were pushed down firmly and sealed with plasticine.

[4] The surgeon's tape used to hold the glass container to the bottom of the bottle was also painted with melted paraffin, for without that treatment it absorbed chlorpicrin very readily.

When all the liquid had evaporated, the cord (b) running from each bottle was pulled in order to remove the stopper from the container (a). Thus the charcoal was not exposed until the gas had reached its full concentration.

As adsorption is very closely related to temperature—the lower the temperature the greater the adsorption—the first work consisted in detemining the adsorption at different known concentrations of chlorpicrin, carbon disulphide, and carbon tetrachloride at temperatures of 15°, 20°, 25°, 30°, and 35° C. The details of these experiments are shown in Tables I to XVI, pages 45 to 59, in the Appendix.

From the data presented in these tables, it was possible to plot the adsorption on 5 grams of active charcoal during one hour against the concentration of the gas and obtain for five different temperatures, covering a range from 15° to 35° C., curves from which unknown concentrations of any one of the three gases could be estimated. These curves are shown in Figures 5, 6, 7, 8, 9, and 10.

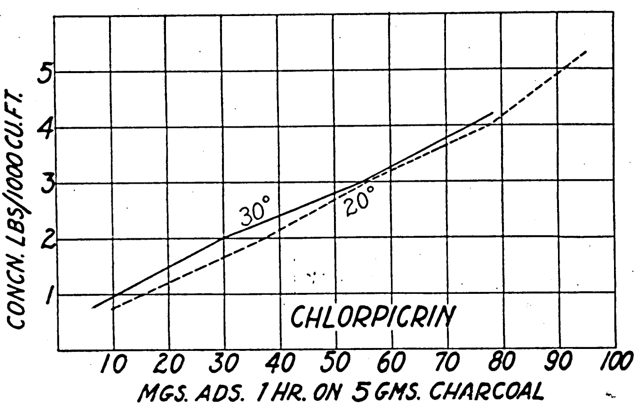

Fig. 5. Adsorption Isotherms for Chlorpicrin Constructed for Estimation of Unknown Concentrations

Experiments on the Diffusion of Chlorpicrin and Carbon Disulphide in a Column of Grain

Equipment Used and Manner of Handling Active Charcoal

In testing the diffusion of chlorpicrin and carbon disulphide in a column of grain, the cylindrical bin (Fig. 11) was used. This was 5 feet high and 14.75 inches in diameter and had a capacity of very slightly less than 6 (5.978) cubic feet. It was constructed of two lengths of glazed sewer pipe placed on a wooden platform. The tile fitted very snugly together, but the narrow cracks between the two tile and between the lower tile and the platform were made air-tight with plasticine. An opening was provided at the bottom of the bin for emptying it, but the trap door for the opening was so built that it could be sealed absolutely tight.

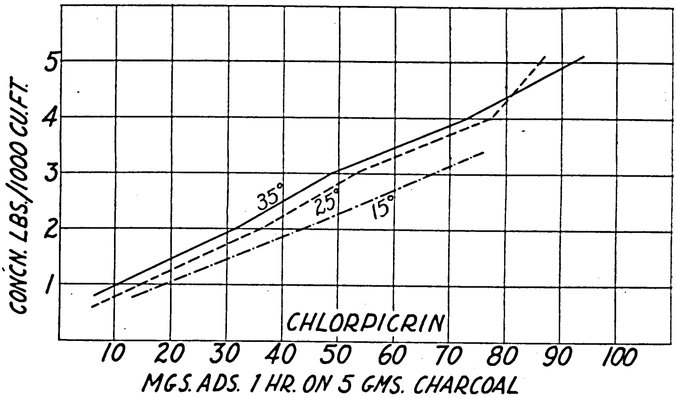

Fig. 6. Adsorption Isotherms for Chlorpicrin Constructed for the Estimation of Unknown Concentrations

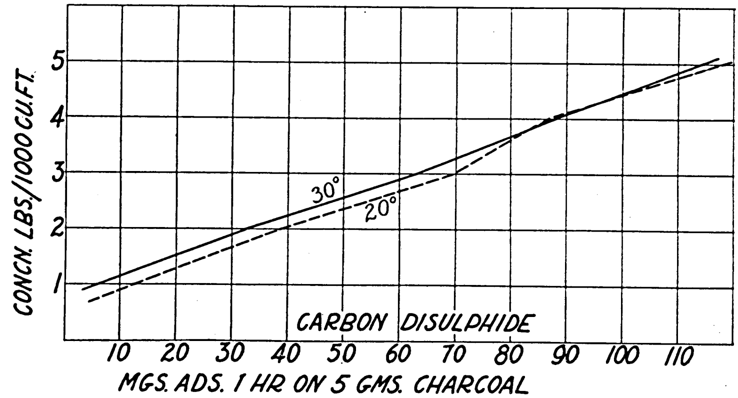

Fig. 7. Adsorption Isotherms for Carbon Disulphide Constructed for the Estimation of Unknown Concentrations

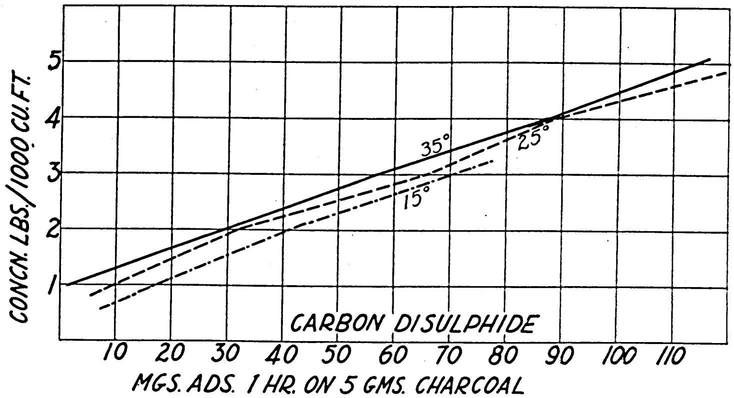

Fig. 8. Adsorption Isotherms for Carbon Disulphide Constructed for the Estimation of Unknown Concentrations

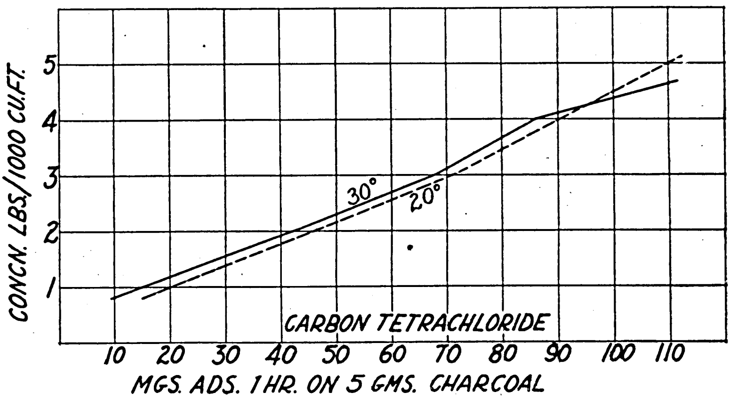

Fig. 9. Adsorption Isotherms for Carbon Tetrachloride Constructed for the Estimation of Unknown Concentrations

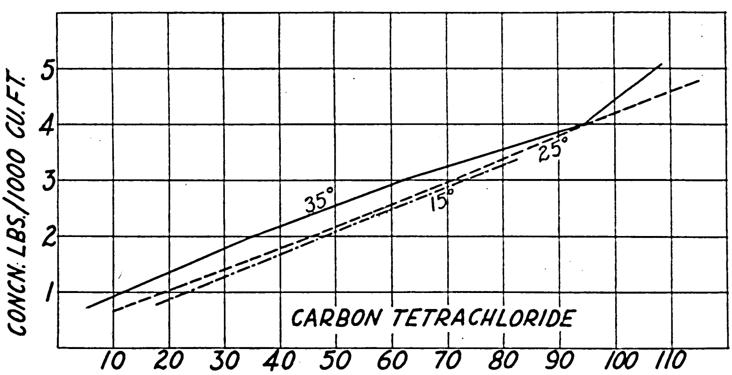

Fig. 10. Adsorption Isotherms for Carbon Tetrachloride Constructed for the Estimation of Unknown Concentrations
The abscissas show milligrams of gas adsorbed on 5 grams of charcoal in one hour.

At the beginning of each experiment in which grain was used, the bin was filled and time was allowed for the grain to settle. Next, the grain was leveled off so that it stood within about 4 inches of the top of the bin. A galvanized iron cover was provided. In this cover were two holes, one large enough for the grain sampler to pass through, and one smaller, through which the stem of a small atomizer, by which the fumigant was applied, could be inserted. After the grain was in the bin, the lid was sealed on. In most of the experiments a test was next made of the adsorption on tubes of charcoal placed in the bin for one hour. Following this check of the adsorption on the charcoal when no fumigant was present, a cover was placed over the hole used for the insertion of the grain sampler and a measured amount of the fumigant was applied.

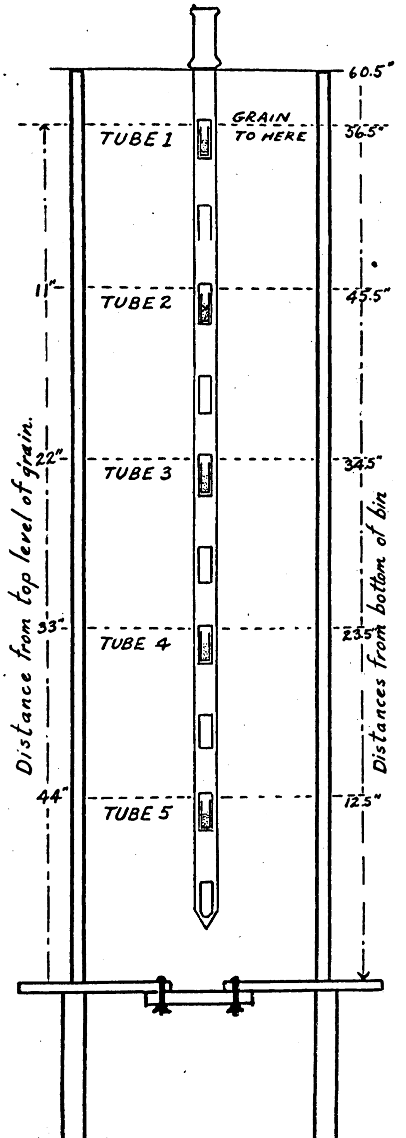

Fig. 11. Diagram of Fumigation Chamber
In this chamber the diffusion of chlorpicrin, carbon disulphide, and carbon tetrachloride in grain was measured by the adsorption of the gases on active charcoal at known depths in a grain sampler.

The use of the grain sampler to lower the tubes of charcoal into the grain is shown in Figure 11. Wire screening was used to prevent the grain from entering the sampler after it was inserted into the grain and opened by revolving the handle. Pieces of screening were cut considerably larger than the openings to the compartments and, when inserted inside, were pressed outward against the inner tube of the sampler. The screens could be pushed up within the compartments far enough to allow the placing or removal of the charcoal tubes.

Test of Apparatus

In order to test out the apparatus, the following preliminary experiment was performed. The trap door at the bottom of the bin was put in place, the top was sealed on with plasticine, and the hole in the top for insertion of the grain sampler was closed. Chlorpicrin at the rate of 3 pounds to 1000 cubic feet was then atomized into the empty bin. After an interval of two hours, tubes of charcoal were placed in the sampler in the positions of tubes 1, 3, and 5, as shown in Figure 11, and the sampler was then lowered into the bin. After an exposure of one hour the tubes were withdrawn and reweighed. The results were as follows:

Temperature, °C.	Position of tube	Weight of charcoal	Increase per 5 gm. charcoal	Approximate concentration per 1000 cu. ft.
		gm.	mg.	lb.
20.5	1	4.9651	18.8	1.17
	3	4.8196	26.6	1.5
	5	4.9974	24.3	1.4

It was immediately seen that the concentration of the gas was not holding up. As the bin was as nearly gas-tight as it could be made, it was thought that adsorption by the tile walls of the bin might account for the loss of the gas. Consequently the bin was shellacked heavily on the inside and the test was repeated. The results follow:

Temperature, °C.	Position of tube	Weight of charcoal	Increase per 5 gm. charcoal	Approximate concentration per 1000 cu. ft.
		gm.	mg.	lb.
20	1	4.9721	52.0	2.8
	2	5.0021	56.3	3.0
	3	5.0110	57.7	3.1

From this it was concluded that the bin was holding the gas satisfactorily.

Diffusion Experiments with Chlorpicrin

Experiment 1.—In the first experiment with grain in the bin, the chlorpicrin was poured directly on the grain instead of being atomized, as in the later experiments. Marquis wheat was used. The chlorpicrin was applied at the rate of 3 pounds per 1000 cubic feet (4.84 cc.). Tests with the charcoal were run during the 2d and 12th hours of the fumigation.

Tube No.	Distance below surface of grain	Weight of charcoal	Increase per 5 gm. charcoal	Maximum concentration per 1000 cu. ft.
	in.	gm.	mg.	lb.
1	0	4.8720	25.3	1.5
2	11	4.8103	4.2	
3	22	4.6920	2.7	Less than 0.4
4	33	4.6739	3.8	
5 (lost)				
Temperature 2d hour of fumigation, 25.5° C.				
1	0	5.0351	7.4	Less than 0.7
2	11	5.0129	6.3	
3	22	5.1469 (spilled)		
4	33	4.8507	6.6	
5	44	5.1109	2.9	
Temperature 12th hour of fumigation, 24.5° C.				

Experiment 2.—Marquis wheat. Chlorpicrin was atomized over the top of the wheat at the rate of 3 pounds per 1000 cubic feet. A tube of charcoal in the position of Tube 3 (Fig. 11), 22 inches below the surface of the wheat, showed previously an increase on 5 grams of 4.1 milligrams. (For an explanation of the value of the check tubes used before fumigations, see the discussion of the results on diffusion.)

Tests were made during the 2d, 5th, 8th, 12th, and 24th hours of the fumigation as follows:

Hour	Temperature, °C.	Distance below surface	Weight of charcoal	Increase per 5 gm. charcoal	Maximum concentration per 1000 cu. ft.
		in.	gm.	mg.	lb.
2d	23.3	0	5.0192	101.6	More than 5
		11	4.9149	25.4	1.5
		22	5.0491	3.2	Less than 0.5
		33	4.8865	3.5	
		44	4.9359	0.9	
5th	26.6	0	4.8001	26.5	1.55
		11	4.7715	21.2	1.3
		22	4.9419	6.9	Less than 0.6
		33	4.8664	6.4	
		44	4.9410	4.1	
8th	26.6	0	5.0598	17.5	1.1
		11	4.8939	14.7	1.0
		22	4.9164	9.0	Less than 0.7
		33	4.9499	8.3	
		44	4.9814	5.9	
12th	26.6	0	4.9893	11.1	0.8
		11	4.8548	8.3	Less than 0.6
		22	4.8188	5.3	
		33	5.0560	6.5	
		44	4.9944 (lost)		
24th	21.6	0	4.7796	6.1	Less than 0.5
		11	4.8445	5.9	
		22	4.9088	2.9	
		33	4.8449	5.2	
		44	4.8410	3.6	

At the completion of the diffusion tests in this experiment, three lots of insects, each containing 10 *Sitophilus oryza* L. and 5 *S. granarius* L. were placed in the positions of tubes 1, 3, and 5, or 0, 22, and 44 inches below the surface of the grain, respectively, for 24 hours. All were killed.

Experiment 3.—In this experiment the weighed amounts of charcoal were put in place in the grain sampler. Then in the usually vacant compartments below those holding the charcoal, five lots of insects (*Tribolium confusum* Duv.) were placed. The sampler was lowered into the grain but before being opened, 3 pounds of chlorpicrin to 1000 cubic feet (4.84 cc.) was applied with the atomizer at the top of the bin. Then the sampler was opened and allowed to remain open for 12 hours. With the charcoal exposed to chlorpicrin for such a length of time we can not estimate the values of the increases on the charcoal. They may be taken, however, to have some relative values as to the distribution of the gas. The results follow:

Temperature, °C.	Distances below surface of grain		Increase per 5 gm. charcoal	Per cent dead
	Charcoal tubes	Insect tubes		
	in.	in.	mg.	
20-25	0*			
	11	16.5	99.0	100
	22	27.5	100.7	100
	33	38.5	82.3	†
	44	49.5	48.1	0

* This tube was omitted on account of high concentration at top of bin.
† Slight movement, all died later.

Experiment 4.—Marquis wheat was in the bin. Chlorpicrin at the rate of 3 pounds to 1000 cubic feet (4.84 cc.) was applied at the top of the bin with an atomizer. Tests on the diffusion of the gas were made during the 2d, 5th, 8th, 12th, and 24th hours. The results were as follows:

Hour	Temperature, °C.	Distance below surface	Weight of charcoal	Increase per 5 gm. charcoal	Maximum concentration per 1000 cu. ft.
		in.	gm.	mg.	lb.
2d	19.5	0	5.1681	102.3	More than 5
		11	5.0846	9.8	Less than 0.7
		22	5.0689	2.8	
		33	5.0085	9.5	
		44	4.9496	7.2	Less than 0.6
5th	20.5	0	5.1240	70.7	3.7
		11	5.0036	20.6	1.2
		22	4.9982	16.5	1.0
		33	4.9719	11.1	0.7
		44	4.9641	9.4	0.6
8th	20.3	0	4.8314	40.2	2.15
		11	4.8396	17.7	1.1
		22	4.8765	11.0	0.7
		33	4.8536	8.0	Less than 0.6
		44	4.8726	7.8	
12th	26.5	0	4.9479	27.5	1.55
		11	5.1064	13.7	0.9
		22	5.0398	10.4	Less than 0.8
		33	5.0182	8.8	
		44	4.9033	9.1	
24th	19.5	0	4.9364	13.7	0.87
		11	4.8156	4.5	Less than 0.5
		22	4.9037	2.8	
		33	5.0322	3.0	
		44	4.9661	1.5	

During the interval between the 12th and 24th hours of the fumigation, a period of 11 hours, open vials containing confused flour beetles (*Tribolium confusum* Duv.) were kept in the positions of 0, 11, 22, 33, and 44 inches below the surface of the grain. On examination immediately after the sampler containing them was withdrawn, those in the upper three vials were dead, those that had been 33 inches below the surface were very evidently affected, but those from 44 inches below the surface were perfectly normal. Forty-eight hours later 40 per cent of the beetles from 33 inches below the surface were dead.

At the beginning of the experiment, a sample of wheat withdrawn from the bin had a moisture content of 11.6 per cent. A sample taken at the conclusion of the experiment showed a moisture content of 10.75 per cent.[5]

Experiment 5.—About 5 pints of wheat were withdrawn from the fumigation bin at the conclusion of Experiment 4. When the wheat was withdrawn and the sampler emptied, the grain from the different compartments was kept separate. The wheat from 1 to 11 inches below the surface was placed in Bottle No. 1, that from 11 to 22 inches in Bottle No. 2, from 22 to 33 inches in Bottle No. 3, from 33 to 44 inches in Bottle No. 4, and from 44 to 55 inches in Bottle No. 5. Before being placed in the bottles the wheat was aired for two hours by being spread out in shallow piles on a table. In each bottle with the wheat were released 10 rice weevils (*Sitophilus oryza* L.) and somewhat later 5 confused flour beetles (*Tribolium confusum* Duv.). A check was kept with wheat which had not been fumigated. After various periods of time the grain in each bottle was aired about 3 hours, the percentage of dead insects was recorded, and then the wheat and fresh live insects were put back in the bottle. The results were as follows:

Bottle No.	Depth in bin	Wheat Withdrawn and Put in Bottles with Insects, August 25, 1924					
		Per cent dead, Aug. 26 (24 hours)		Per cent dead, Aug. 28 (72 hours)		Per cent dead, Aug. 30 (5 days)	
		Sitophilus	Tribolium*	Sitophilus	Tribolium	Sitophilus	Tribolium
	in.						
1	0-11	100	...	100	100	100	100
2	11-22	100	...	100	60	100	20
3	22-33	100	...	100	20	100	0
4	33-44	100	...	100	0	50	0
5	44-55	100	...	50	0	20	0
Check		0	...	0	0	0	0

Bottle No.	Depth in bin	Per cent dead, Sept. 6 (12 days)		Per cent dead, Sept. 10 (16 days)	
		Sitophilus	Tribolium	Sitophilus	Tribolium
	in.				
1	0-11	100	100	80	0
2	11-22	100	40	60	0
3	22-33	100	20	20	0
4	33-44	100	0	20	0
5	44-55	100	0	30	0
Check		0	0	0	0

* Put in on Aug. 26.

The experiment shows very plainly that chlorpicrin must have been taken up by the wheat in considerable quantity to kill 80 per cent of the weevils 16 days after being fumigated and after a total of 14

[5] The writer is indebted to the Division of Agricultural Biochemistry for the determinations of moisture content.

hours airing. Even tho *Sitophilus oryza* L. is undoubtedly more susceptible to chlorpicrin than *Tribolium confusum* Duv., the much greater mortality of the *Sitophilus* in this experiment may have been due to their feeding on the grain, whereas *Tribolium* did not. The fact that *Tribolium* were killed shows that chlorpicrin, as a gas, was present in the bottles. The odor of chlorpicrin was very evident in the bottles holding samples of wheat taken from near the top of the bin.

Diffusion Experiments with Carbon Disulphide

Experiment 6.—The experiments with carbon disulphide were performed with the same kind of wheat as was used in the chlorpicrin experiments. Carbon disulphide was used in the fumigation bin at the rate of 3 pounds to 1000 cubic feet, or 6.5 cc. This low concentration was used in order to observe more closely the diffusion of the gas. Tests for diffusion were made during the 2d, 5th, and 24th hours. Before the application of the disulphide, a test was made of the adsorption of moisture by the charcoal.

Check on Adsorption of Moisture

Hour	Temperature, °C.	Distance below surface	Weight of charcoal	Increase per 5 gm. charcoal	Maximum concentration per 1000 cu. ft.
		in.	gm.	mg.	lb.
	22.5	0	5.1854	3.0	
		11	5.0621	6.2	
		22	5.0290	5.9	
		33	5.0335	8.5	
		44	5.1160	6.3	
2d	22.5	0	5.1884	158.4	More than 5
		11	5.0683	24.8	1.5
		22	5.0359	3.7	Less than 0.7
		33	5.0420	2.8	
		44	5.0420	3.1	
5th	24.5	0	5.0317	68.8	3.12
		11	4.9815	30.8	2.0
		22	4.8654	15.3	1.27
		33	4.9775	12.3	1.12
		44	4.8967	12.3	1.12
24th	27	0	4.9018	16.1	1.3
		11	4.7470	13.0	1.2
		22	4.8611	10.1	1.05
		33	5.0957	10.4	1.06
		44	5.0750	8.6	0.95

Experiment 7.—Carbon disulphide was used at the rate of 3 pounds to 1000 cubic feet, or 6.5 cc., in the fumigation bin. Marquis wheat was used. Tests for diffusion were made at the 2d, 5th, and 24th hours. Check on moisture, etc., was made first.

Check on Adsorption of Moisture

Hour	Temperature, °C.	Distance below surface	Weight of charcoal	Increase per 5 gm. charcoal		Maximum concentration per 1000 cu. ft.
		in.	gm.	mg.		lb.
	25	0	5.0025	6.0		
		11	5.0047	6.2		
		22	5.0010	6.4		
		33	5.0018	6.8		
		44	5.0010	5.7		
2d	25	0	5.0035	93.6		4.15
		11	5.0011	16.9		1.35
		22	5.0053	16.1		1.3
		33	4.9983	10.4		1.07
		44	4.9987	5.1	Less than	0.9
5th	26	0	5.0019	56.2		2.75
		11	4.9926	19.6		1.47
		22	5.0002	10.6		1.07
		33	5.0011	6.7	Less than	0.9
		44	4.9984	7.4		
24th	26	0	5.0000	9.8		1.03
		11	5.0027	9.1		1.0
		22	5.0024	8.3		
		33	4.5999	2.3	Less than	0.9
		44	5.0011	3.3		

Diffusion Experiments with Carbon Tetrachloride

The following experiment was performed with carbon tetrachloride in the diffusion chamber at the rate of three pounds per 1000 cubic feet. As near as could be determined the wheat was similar to that used in former experiments.

Check on Adsorption of Moisture

Hour	Temperature, °C.	Distance below surface	Weight of charcoal	Increase per 5 gm. charcoal		Maximum concentration per 1000 cu. ft.
		in.	gm.	mg.		lb.
	23	0	5.0062	7.0		
		11	5.0107	5.0		
		22	5.0009	8.2		
		33	5.0062	6.5		
		44	5.0092	6.2		
2d	23	0	5.0065	78.4		3.3
		11	4.9880	33.2		1.5
		22	5.0101	34.1		1.5
		33	5.0046	18.3		1.0
		44	5.0092	10.4	Less than	0.8
5th	22	0	5.0007	34.3		1.5
		11	4.9958	18.2		1.0
		22	5.0017	12.9		
		33	5.0062	15.1	Less than	1.0
		44	5.0102	14.0		
12th	22	0	4.9890	10.2		
		11	5.0020	14.0	Less than	1.0
		22	5.0100	9.5		
		33	5.0062	10.1		
		44	5.0020	8.7		
24th	23	0	5.0091	6.1		
		11	5.0076	12.0		
		22	4.9992	9.2	Less than	1.0
		33	4.9982	7.1		
		44	5.0009	10.2		

As the concentration of the fumigant used in the previous experiment was far below what would be toxic to insects, in another experiment the wheat in the fumigation chamber was treated with carbon tetrachloride at the rate of 30 pounds per 1000 cubic feet. The adsorption on the charcoal shows at least the relative concentrations at the different depths. The amount of gas adsorbed during the fifth hour was so small that no further determinations were made.

Hour	Temperature, °C.	Distance below surface	Weight of charcoal	Increase per 5 gm. charcoal	Approximate maxmium concentration per 1000 cu. ft.
		in.	gm.	mg.	lb.
2d	24	0	5.0069	58.3	2.5
		22	4.9985	143.5	
		44	4.9976	126.4	
5th	24	0	5.0041	21.0	1.1
		22	5.0094	13.0	Less than 0.8
		44	4.9992	26.0	1.3

Discussion of Results of Diffusion Experiments

Probable error in the use of active charcoal.—Whether the increases in weight were due to moisture or to the adsorption of the fumigant could not be determined in the tubes of charcoal showing a small concentration. However, even with some water vapor taken up, the increases on the charcoal would show maximum concentrations. Cheney, Ray, and St. John (1923) have shown that active charcoal possesses the quality of selectivity. For instance, when it is added to a mixture of water and benzene, it accepts the benzene but rejects the water. Whether or not this is true of the adsorption of these gases in preference to water vapor we do not know.

General results.—The gases undoubtedly diffuse downward, but comparatively slowly at ordinary temperatures. There is little foundation, so far as we have determined, for the statement that because the vapors formed by the evaporation of such substances as chlorpicrin or carbon disulphide are several times heavier than air, they therefore always will sink rapidly to the bottom of a bin containing cereal grain.

The concentration of carbon disulphide in a tight bin covered at the top is not greater at the bottom of the bin than toward the top. On the contrary, the concentration of the gas at the end of the 2d, 5th, or 24th hour of the fumigation is inversely proportional to the depth below the surface of the grain.

The experiments on carbon tetrachloride show a stronger tendency for a downward movement of the gas than was the case with chlorpicrin or carbon disulphide. But what is perhaps even more important, the second experiment gives an idea of the great amount of fumigant taken up by the grain.

The diffusion downward seems to be closely related to the adsorption of the fumigant by the grain. Theoretically, the higher the temperature the less the adsorption, consequently there should be a greater tendency for the fumigant to diffuse through the entire mass of grain. On the other hand, the lower the temperature the greater the adsorption, and the tendency for the gas to diffuse would be diminished.

Table III shows the relation of temperature to the adsorption of chlorpicrin, carbon disulphide, and carbon tetrachloride on active charcoal.

TABLE III

QUANTITY ADSORBED BY 5 GRAMS OF ACTIVE CHARCOAL IN ONE HOUR
(Concentration 1 Pound to 1000 Cubic Feet)

Temperature, °C.	CS_2 mg.	CCl_3NO_2 mg.	CCl_4 mg.
35	1.8	10.3	11.8
30	5.7	10.6	14.9
25	9.2	14.7	19.6
20	12.4	15.6	20.2
15	17.1	18.9	22.0

These data are shown in Figure 12. It will be seen that at 15° C. 9.5 times more carbon disulphide was adsorbed than at 35°, about 1.8 times as much chlorpicrin, and nearly twice as much carbon tetrachloride. The amount of adsorbent used, 10 cc., was only one eight hundred sixty-fifth of the volume of the container. From these data it seems probable that altho wheat has unquestionably a very small fraction of the adsorption power of active charcoal, when a bin is full of wheat and a fumigant is applied, the wheat, as the adsorbent, is

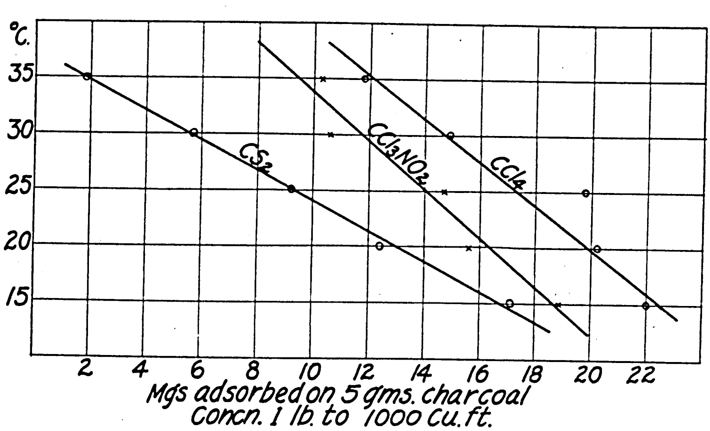

Fig. 12. Curves Showing the Relation of Temperature to the Adsorption of Chlorpicrin, Carbon Disulphide, and Carbon Tetrachloride on Active Charcoal

affected by temperature in some such ratio. The temperature used in the fumigation of grain—merely from the standpoint of diffusion—seems to be particularly important in the case of carbon disulphide. Added weight is given to this viewpoint by the fact that carbon disulphide maintains its toxicity to insects at fairly low temperature, as previously shown.

Hinds (1917) is probably correct in his explanation regarding the effectiveness of carbon disulphide when applied to large masses of corn. From what we have found, the fumigation of such large piles of unbinned corn for protection against weevil should be most successful at lower temperatures, that is, temperatures from 60° to 70° F. rather than above 80° F.

CONCLUSIONS

The toxicity of chlorpicrin, carbon disulphide, and carbon tetrachloride to insects can be correctly compared only when concentration, time to kill, and temperature are all considered as factors.

Throughout a range of temperatures from 35° to 10° C., 1 pound of chlorpicrin is more toxic to the confused flour beetle (*Tribolium confusum* Duv.) than 20 pounds of carbon tetrachloride, and the lower the temperature the greater the relative toxicity. Throughout the same range of temperatures, chlorpicrin at the rate of 1 pound to 1000 cubic feet of space is less toxic than carbon disulphide at the rate of 15 pounds per 1000 cubic feet.

The greater the concentration of chlorpicrin (between 1 and 3 pounds per 1000 cubic feet) the better is its toxicity to insects maintained through a range in temperatures from 35° to 10° C.

A decrease in temperature lowers the toxicity of carbon tetrachloride much more rapidly than that of carbon disulphide or chlorpicrin.

When time to kill 100 per cent of *Tribolium confusum* Duv. with chlorpicrin, carbon disulphide, or carbon tetrachloride is plotted against temperature, the result is not a straight line. Between 35° and 10° C. the time increases at a greater rate toward the lower temperatures.

When chlorpicrin or carbon disulphide is applied to wheat in a tight bin, the downward diffusion of the gas is not so rapid as ordinarily described.

The concentration of the gases does not become stronger at the bottom of a bin of wheat than toward the top, but varies inversely with the depth below the surface of the grain.

Adsorption of the gases by the top layers of grain prevents their rapid downward movement.

As adsorption is closely related to temperature (the lower the temperature the greater the adsorption) temperature is an important factor in the fumigation of grain aside from its relation to the toxicity of the fumigant or the activity of the insects being fumigated.

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APPENDIX

Experiments with Chlorpicrin

(See page 10.)

Experiment 1. Concentration 1 lb. to 1000 cu. ft. Temperature 35° C. R. H. 50%
CCl_3NO_2 Applied 7:59 p.m. Insects exposed at 8:10 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	9:00	45	0
2	9:05	55	0
3	9:10	1:00	60
4	9:15	1:05	60
5	9:25	1:15	100
6	9:35	1:25	100
7	9:40	1:30	100
8	9:45	1:35	100
9	9:50	1:40	100
10	9:55	1:45	100

The *Tribolium* in the observation vial were still very active at the end of 50 minutes.

Experiment 2. Concentration 2 lb. to 1000 cu. ft. Temperature 35° C. R. H. 50%
CCl_3NO_2 applied 9:25 a.m. Insects exposed at 9:40 a.m.

Cage No.	Taken out, a.m.	Exposure	% Dead
		min.	
1	9:55	15	20
2	9:59	19	60
3	10:03	23	60
4	10:07[1]	27	20
5	10:11	31	100
6	10:15	35	100
7	10:19	39	100
8	10:23	43	100
9	10:27	47	100
10	10:31	51	100

Experiment 3. Concentration 3 lb. to 1000 cu. ft. Temperature 35° C. R. H. 50%
CCl_3NO_2 applied 1:50 p.m. Insects exposed at 2:02 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		min.	
1	2:05	3	0
2	2:08	6	0
3	2:11	9	20
4	2:14	12	80
5	2:17	15*	100
6	2:20	18	80
7	2:23	21*	100
8	2:26	24	100
9	2:29	27	100
10	2:32	30	100

[1] In cases such as this both times were recorded on the graph showing comparative toxicities. See Figure 2.

Experiment 4. Concentration 1 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%
CCl_3NO_2 applied 3:52 p.m. Insects exposed at 4:05 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	4:30	25	20
2	4:40	35	20
3	4:50	45	50
4	5:00	55	60
5	5:10	1:05	60
6	5:20	1:15	70
7	5:30	1:25	90
8	5:40	1:35	80
9	5:50	1:45	100
10	6:00	1:55	100

In this experiment as well as in experiments 5, 6, and 7 ten insects were in each cage.

Experiment 5. Concentration 2 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%
CCl_3NO_2 applied 3:45 p.m. Insects exposed at 4:05 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		min.	
1	4:15	10	10
2	4:20	15	20
3	4:25	20	10
4	4:30	25	30
5	4:35	30	60
6	4:40	35	100
7	4:45	40	90
8	4:50	45	100
9	4:55	50	100
10	5:00	55	100

Experiment 6. Concentration 3 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%
CCl_3NO_2 applied 8:40 p.m. Insects exposed at 8:51 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		min.	
1	8:56	5	0
2	9:01	10	60
3	9:06	15	70
4	9:11	20	40
5	9:16	25	100
6	9:21	30	100
7	9:26	35	100
8	9:31	40	100
9	9:36	45	100
10	9:41	50	100

Experiment 7. Concentration 4 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%
CCl_3NO_2 applied 3:29 p.m. Insects exposed at 3:48 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		min.	
1	3:52	4	50
2	3:56	8	40
3	4:00	12	90
4	4:04	16	100
5	4:08	20	100
6	4:12	24	100
7	4:16	28	100
8	4:20	32	100
9	4:24	36	100
10	4:25	40	100

Experiment 8. Concentration 1 lb to 1000 cu. ft. Temperature 25° C. R. H. 50%
CCl_3NO_2 applied 4:45 p.m. Insects exposed at 5:00 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	6:15	1:15	20
2	6:45	1:45	80
3	7:15	2:15	100
4	7:25	2:25	100
5	7:35	2:35	100
6	7:40	2:40	100
7	7:45	2:45	100
8	7:50	2:50	100
9	7:55	2.55	100
10	8:00	3:00	100

Experiment 9. Concentration 2 lb. to 1000 cu. ft. Temperature 25° C. R. H. 50%
CCl_3NO_2 applied 9:45 p.m. Insects exposed at 10 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	10:30	30	0
2	10:40	40	80
3	10:50	50	100
4	11:00	1:00	100
5	11:05	1:05	100
6	11:10	1:10	100
7	11:15	1:15	100
8	11:20	1:20	100
9	11:25	1:25	100
10	11:30	1:30	100

Experiment 10. Concentration 3 lb. to 1000 cu. ft. Temperature 25° C. R. H. 50%
CCl_3NO_2 applied 1:05 a.m. Insects exposed at 1:30 a.m.

Cage No.	Taken out, a.m.	Exposure	% Dead
		min.	
1	1:40	10	0
2	1:45	15	40
3	1:50	20	80
4	1:55	25	60
5	2:00	30	100
6	2:05	35	100
7	2:10	40	100
8	2:15	45	100
9	2:20	50	100
10	2:25	55	100

Experiment 11. Concentration 1 lb. to 1000 cu. ft. Temperature 20° C. R. H. 50%
CCl_3NO_2 applied 5:30 p.m. Insects exposed at 6:00 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	8:15	2:15	80
2	8:30	2:30	80
3	8:45	2:45	100
4	9:00	3:00	100
5	9:15	3:15	100
6	9:30	3:30	100
7	9:45	3:45	100
8	10:00	4:00	100
9	10:15	4:15	100
10	10:30	4:30	100

Experiment 12. Concentration 2 lb. to 1000 cu ft. Temperature 20° C. R. H. 50%
CCl_3NO_2 applied 11:45 a.m. Insects exposed at 12:15 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	12:55	40	0
2	1:05	50	40
3	1:10	55	20
4	1:15	1:00	60
5	1:20	1:05	80
6	1:25	1:10	100
7	1:30	1:15	100
8	1:35	1:20	100
9	1:40	1:25	100
10	1:45	1:30	100

Experiment 13. Concentration 3 lb. to 1000 cu. ft. Temperature 20° C. R. H. 50%
CCl_3NO_2 applied 12:15 a.m. Insects exposed at 12:45 a.m.

Cage No.	Taken out, a.m.	Exposure	% Dead
		hr.-min.	
1	1:10	25	0
2	1:15	30	80
3	1:20	35	80
4	1:25	40	100
5	1:30	45	100
6	1:35	50	100
7	1:40	55	100
8	1:45	1:00	100
9	1:50	1:05	100
10	1:55	1:10	100

Experiment 14. Concentration 1 lb. to 1000 cu ft. Temperature 15° C. R. H. 50%
CCl_3NO_2 applied 10:00 a.m. Insects exposed at 10:25 a.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	12:30	2:05	40
2	1:00	2:35	60
3	1:15	2:50	60
4	1:30	3:05	80
5	1:45	3:20	60
6	2:00	3:35	80
7	2:15	3:50	100
8	2:30	4:05	100
9	2:45	4:20	100
10	3:00	4:35	100

Experiment 15. Concentration 2 lb. to 1000 cu. ft. Temperature 15° C. R. H. 50%
CCl_3NO_2 applied 6:10 p.m. Insects exposed at 9:10 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	10:15	1:05	20
2	10:30	1:20	80
3	10:40	1:30	80
4	10:50	1:40	80
5	11:00	1:50	80
6	11:10	2:00	100
7	11:20	2:10	100
8	11:30	2:20	100
9	11:40	2:30	100
10	11:50	2:40	100

Experiment 16. Concentration 3 lb. to 1000 cu. ft. Temperature 15° C. R. H. 50%
CCl_3NO_2 applied 12:10 p.m. Insects exposed at 1:50 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min	
1	2:20	30	0
2	2:25	35	20
3	2:30	40	20
4	2:35	45	40
5	2:40	50	20
6	2:45	55	80
7	2:50	1:00	100
8	2:55	1:05	100
9	3:00	1:10	100
10	3:05	1:15	100

Experiment 17. Concentration 1 lb. to 1000 cu. ft. Temperature 10° C. R. H. 50%
CCl_3NO_2 applied 6:15 p.m. Insects exposed at 6:45 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	8:45	2:00	0
2	9:45	3:00	0
3	10:15	3:30	40
4	10:45	4:00	40
5	11:00	4:15	80
6	11:15	4:30	100
7	11:30	4:45	100
8	11:45	5:00	100
9	12:00 m.	5:15	100
10	12:15 a.m.	5:30	100

Experiment 18. Concentration 2 lb. to 1000 cu. ft. Temperature 10° C. R. H. 50%
CCl_3NO_2 applied 8:30 p.m. Insects exposed at 9 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	10:30	1:30	
2	11:00	2:00	80
3	11:30	2:30*	80
4	11:40	2:40	100
5	11:50	2:50	100
6	12:00	3:00	100
7	12:10	3:10	100
8	12:20	3:20	100
9	12:30	3:30	100
10	12:40	3:40	100

* One beetle was particularly active here immediately after the cage containing it was withdrawn from the fumigation jar. Those in cage No. 2 were all active when withdrawn.

Experiment 19.* Concentration 3 lb. to 1000 cu. ft. Temperature 10° C. R. H. 50%
CCl_3NO_2 applied 12:55 a.m. Insects exposed at 1:20 a.m.

Cage No.	Taken out, a.m.	Exposure	% Dead
		hr.-min.	
1	2:00	40	40
2	2:10	50	60
3	2:20	1:00	..
4	2:30	1:10	80
5	2:35	1:15	80
6	2:40	1:20	100
7	2:45	1:25	100
8	2:50	1:30	100
9	2:55	1:35	100
10	3:00	1:40	100

* On account of the insects being left in the cages for several hours following the completion of this experiment and the fact that the smell of chlorpicrin seemed to be rather strong on the cages when examined, another trial was made. However, the second trial gave almost identical results.

Experiments with Carbon Disulphide

Experiment 20. Concentration 15 lb. to 1000 cu. ft. Temperature 35° C. R. H. 50%
CS_2 applied 9:45 p.m. Insects exposed at 9:55 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		min.	
1	10:00	5	0
2	10:05	10	0
3	10:10	15	20
4	10:15	20	40
5	10:20	25	80
6	10:25	30	100
7	10:30	35	100
8	10:35	40	100
9	10:40	45	100
10	10:45	50	100

Experiment 21.* Concentration 10 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%
CS_2 applied 9:10 p.m. Insects exposed at 9:15 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	10:00	45	80
2	10:15	60	69
3	10:25	1:10	80
4	10:35	1:20	50
5	10:45	1:30	100
6	10:55	1:40	100
7	11:00	1:45	100
8	11:05	1:50	100
9	11:10	1:55	100
10	11:15	2:00	100

* On account of the high percentage of kill, even at 45 minutes, in this experiment it was repeated as follows:

CS_2 applied at 11:15 a.m. Insects exposed at 11:20 a.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	12:05	45	40
2	12:15	55	80
3	12:25	1:05	100
4	12:35	1:15	100

One hundred per cent was killed in the six other cages taken out at intervals up to 1 hour and 50 minutes. The results of both these trials are recorded on Figure 3.

Experiment 22. Concentration 15 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%
CS_2 applied at 9:25 p.m. Insects exposed at 9:30 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		min.	
1	9:40	10	0
2	9:45	15	80
3	9:50	20	20
4	9:55	25*	100
5	10:00	30	60
6	10:05	35	60
7	10:10	40	80
8	10:15	45	100
9	10:20	50	100
10	10:25	55	100

* Both of these periods are recorded on Figure 2.

Experiment 23. Concentration 20 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%
CS_2 applied 2:27 p.m. Insects exposed at 2:33 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		min.	
1	2:43	10	60
2	2:50	17	40
3	2:55	22	80
4	3:00	27	100
5	3:05	32	100
6	3:10	37	100
7	3:15	42	100
8	3:20	47	100
9	3:25	52	100
10	3:30	57	100

Experiment 24. Concentration 15 lb. to 1000 cu. ft. Temperature 25° C. R. H. 50%
CS_2 applied 9:15 p.m. Insects exposed at 9:30 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	10:00	30	0
2	10:10	40	20
3	10:20	50	60
4	10:30	1:00*	100
5	10:40	1:10	100
6	10:50	1:20	80
7	11:00	1:30*	100
8	11:10	1:40	100
9	11:20	1:50	100
10	11:30	2:00	100

* Both these periods are recorded on Figure 2.

Experiment 25.* Concentration 15 lb. to 1000 cu. ft. Temperature 20° C. R. H. 50%
CS_2 applied 4:00 a.m. Insects exposed at 4:00 a.m.

Cage No.	Taken out, a.m.	Exposure	% Dead
		hr.-min.	
1	5:30	1:30	60
2	6:00	2:00	100
3	6:30	2:30	100
4	7:00	3:00	100
5	7:30	3:30	100
6	8:00	4:00	100
7	10:00	6:00	100
8	11:00	7:00	100
9	12:00 m.	8:00	100
10	1:00 p.m.	9:00	100

* This experiment was performed in a large wide-mouthed bottle, 8650 cc. capacity. The evaporation of CS_2 is so rapid that the short time that the insects were exposed before the gas was full strength would make little, if any, difference. The other apparatus was in use at the same time.

Experiment 26. Concentration 15 lb. to 1000 cu. ft. Temperature 15° C. R. H. 50%
CS_2 applied and insects exposed at 11:50 a.m.
(Performed in the same container as Experiment 25.)

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	1:50	2:00	40
2	2:50	3:00	100
3	3:50	4:00	100
4	4:50	5:00	100
5	5:50	6:00	100
6	6:50	7:00	100
7	7:50	8:00	100

Experiment 27. Concentration 15 lb. to 1000 cu. ft. Temperature 10° C. R. H. 50%
CS_2 applied and insects exposed at 8:30 p.m.
(The same container was used as in the two previous experiments.)

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	10:00	1:30	0
2	10:30	2:00	0
3	11:00	2:30	0
4	11:30	3:00	40
5	11:40	3:10	60
6	11:50	3:20	40
7	12:00 m.	3:30	100
8	12:10 a.m.	3:40	100
9	12:20	3:50	100
10	12:30	4:00	100

Experiments with Carbon Tetrachloride

Experiment 28. Concentration 20 lb. to 1000 cu. ft. Temperature 35° C. R. H. 50%
CCl_4 applied 3:50 p.m. Insects exposed at 4:06 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	5:36	1:30	100
2	5:45	1:39	100
3	5:54	1:48	100

Cages 4 to 10, taken out at intervals up to 2:51, showed 100 per cent kill. For this reason the experiment was repeated and the cages were taken out at shorter periods of time. The results follow:

CCl_4 applied at 10:04 a.m. Insects exposed at 10:15 a.m.

Cage No.	Taken out, a.m.	Exposure	% Dead
		hr.-min.	
1	10:35	20	0
2	10:45	30	20
3	10:55	40	0
4	11:00	45	20
5	11:05	50	0
6	11:10	55	0
7	11:15	1:00	20
8	11:20	1:05	0
9	11:25	1:10	0
10	11:30	1:15	0

It is evident that the real minimum of time to kill was missed in both of these experiments. One hour and 30 minutes, the time obtained from the first part of this experiment, was used on Figure 2.

Experiment 29. Concentration 1 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%

Insects were exposed as in the other experiments for periods up to 5 hours and 20 minutes. Not a single specimen died.

Experiment 30. Concentration 4 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%

The insects were exposed tc the fumigant as in the other experiments for periods up to 4 hours and 15 minutes. Not a single specimen died.

Experiment 31. Concentration 10 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%

The insects were exposed to the fumigant for periods up to 7 hours. Altho they all became inactive in less than 2 hours, when the first cage was removed, none were killed up to 4 hours and 30 minutes and only 20% in 7 hours.

Experiment 32. Concentration 20 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%
CCl_4 applied 10:14 a.m. Insects exposed at 10:50 a.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	11:45 a.m.	1:15	40
2	12:00 m.	1:30	60
3	12:15	1:45	20
4	12:30	2:00	40
5	12:45	2:15	40
6	1:00	2:30	60
7	1:15	2:45	80
8	1:30	3:00	80
9	1:45	3:15	100
10	2:00	3:30	100

Experiment 33. Concentration 30 lb. to 1000 cu. ft. Temperature 30° C. R. H. 50%
CCl_4 applied 1:33 p.m. Insects exposed at 1:45 p.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	2:45	1:00	60
2	3:00	1:15	0
3	3:15	1:30	80
4	3:30	1:45	60
5	3:45	2:00	100
6	4:00	2:15	100
7	4:15	2:30	80
8	4:30	2:45	60
9	4:45	3:00	100
10	5:00	3:15	100

Experiment 34. Concentration 20 lb. to 1000 cu. ft. Temperature 25° C. R. H. 50%
CCl_4 applied 11:25 a.m. Insects exposed at 11:45 a.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	1:15	1:30	0
2	1:35	1:50	0
3	1:50	2:05	20
4	2:05	2:20	0
5	2:20	2:35	20
6	2:35	2:50	0
7	2:55	3:10	20
8	3:05	3:20	20
9	3:20	3:35	40
10	3:30	3:45	60

As a 100% kill was not obtained, the experiment was repeated as follows:
CCl_4 applied 11:25 a.m. Insects exposed at 11:45 a.m.

Cage No.	Taken out, p.m.	Exposure	% Dead
		hr.-min.	
1	2:45	3:00	20
2	3:05	3:20	40
3	3:25	3:40	40
4	3:45	4:00	40
5	4:05	4:20	40
6	4:25	4:40	60
7	4:45	5:00	60
8	5:05	5:20	80
9	5:25	5:40	60
10	5:45	6:00	60
11	6:45	7:00	100
12	7:45	8:00	100

Experiment 35. Concentration 20 lb. to 1000 cu. ft. Temperature 20° C. R. H. 50%
CCl_4 applied 3:45 a.m. Insects exposed at 4:00 a.m.

Cage No.	Taken out, a.m.	Exposure	% Dead
		hr.-min.	
1	7:00	3:00	40
2	8:00	4:00	20
3	9:00	5:00	..*
4	10:00	6:00	..
5	11:00	7:00	..
6	12:00 m.	8:00	..
7	1:00 p.m.	9:00	..
8	2:00	10:00	..
9	3:00	11:00	..
10	4:00	12:00	100?
11	5:00	13:00	100
12	6:00	14:00	100
13	7:00	15:00	100
14	8:00	16:00	100

* It was very difficult to tell whether the insects taken from cages 3 to 9 were dead or alive. At the end of 24 hours most of them showed some movement of appendages but so slight that it was impossible to say just where the minimum time to kill 100% occurred.

TABLE I

Gas Adsorbed, Chlorpicrin; Temperature, 35° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6886	4.9984	29.6990	0.0104	10.4	9.7
	2	21.9520	26.9571	5.0051	26.9655	.0084	8.4	
	3	22.0357	27.0484	5.0127	27.0586	.0102	10.2	
2	1	24.6902	29.6927	5.0025	29.7270	.0343	34.3	31.3
	2	21.9520	26.9589	5.0069	26.9917	.0326	32.7	
	3	22.0357	27.0306	4.9949	27.0576	.0270	27.0	
3	1	24.6902	29.6862	4.9960	29.7377	.0514	51.4	48.1
	2	21.9520	26.9652	5.0132	27.0132	.0480	47.9	
	3	22.0357	27.0343	4.9986	27.0794	.0451	45.1	
4	1	24.6902	29.6980	5.0078	29.7734	.0754	75.3	72.5
	1	......	29.6871	4.9969	29.7567	.0696	69.7	
5	2	21.9520	26.9671	5.0151	27.0584	.0913	91.0	91.0
Check	3	22.0357	27.0430	5.0083	27.0377	—.0053	—5.3	—3.6
	3	......	27.0280	4.9923	27.0261	—.0019	—1.9	

TABLE II

Gas Adsorbed, Chlorpicrin; Temperature, 30° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6696	5.0094	29.7100	0.0104	10.4	10.6
	2	21.9520	26.9632	5.0112	26.9748	.0116	11.6	
	3	22.0357	27.0307	4.9950	27.0403	.0096	9.6	
	1	24.6902	29.6835	4.9933	29.6936	.0101	10.1	
	2	21.9520	26.9583	5.0063	26.9706	.0123	12.3	
	3	22.0357	27.0247	4.9890	27.0347	.0100	10.0	
2	1	24.6902	29.6936	5.0034	29.7249	.0313	31.3	29.7
	2	21.9520	26.9570	5.0050	26.9851	.0281	28.1	
	3	22.0357	27.0318	4.9961	27.0615	.0297	29.7	
3	1	24.6902	29.6950	5.0048	29.7552	.0602	60.1	55.1
	2	21.9520	26.9629	5.0109	27.0153	.0524	52.3	
	3	22.0357	27.0322	4.9965	27.0851	.0529	52.9	
4	1	24.6902	29.6947	5.0045	29.7700	.0744	74.3	74.3
5	2	21.9520	26.9605	5.0085	27.0384	.0779	77.7*	77.7
Check	3	22.0357	27.0370	5.0013	27.0376	0.0006	0.6	0.6

* Evidently not all of the chlorpicrin was evaporated when the charcoal was exposed.

TABLE III

Gas Adsorbed, Chlorpicrin; Temperature, 25° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6890	4.9988	29.7036	0.0146	14.6	14.7
	2	21.9520	26.9568	5.0048	26.9700	.0132	13.2	
	3	22.0357	27.0322	4.9965	27.0485	.0163	16.3	
2	1	24.6902	29.6952	5.0050	29.7314	.0362	36.1	36.4
	2	21.9520	26.9564	5.0044	26.9952	.0392	39.1	
	3	22.0357	27.0352	4.9995	27.0693	.0341	34.1	
3	1	24.6902	29.6880	4.9978	29.7396	.0516	51.6	52.4
	2	21.9520	26.9535	5.0015	27.0093	.0558	55.8	
	3	22.0357	27.0330	4.9973	27.0830	.0500	50.0	
4	1	24.6902	29.6917	5.0015	29.7690	.0773	77.3	77.3
5	2	21.9520	26.9589	5.0069	27.0450	.0861	85.9	85.9
Check	3	22.0357	27.0358	5.0001	27.0383	0.0025	2.5	2.5

TABLE IV

Gas Adsorbed, Chlorpicrin; Temperature, 20° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6890	4.9988	29.7047	0.0157	15.7	15.6
	2	21.9520	26.9562	5.0042	26.9711	.0149	14.9	
	3	22.0357	27.0318	4.9961	27.0479	.0161	16.1	
2	1	24.6902	29.6882	4.9980	29.7252	.0370	37.0	37.2
	2	21.9520	26.9607	5.0087	26.9973	.0366	36.5	
	3	22,0357	27.0367	5.0010	27.0748	.0381	38.1	
3	1	24.6902	29.6923	5.0021	29.7454	.0531	53.1	56.0
	2	21.9520	26.9530	5.0010	27.0086	.0556	55.6	
	3	22.0357	27.0358	5.0001	27.0950	.0592	59.2	
4	1	24.6902	29.6910	5.0008	29.7689	.0779	77.9	77.9
5	2	21.9520	26.9554	5.0034	27.0469	.0915	91.4	91.4
Check	3	22.0357	27.0375	5.0018	27.0435	0.0060	6.0	6.0

TABLE V

Gas Adsorbed, Chlorpicrin; Temperature, 15° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6865	4.9953	29.7064	0.0199	19.9	18.9
	1	......	29.6894	4.9992	29.7080	.0186	18.6	
	1	......	29.6917	5.0015	29.7099	.0182	18.2	
2	2	21.9520	26.9545	5.0025	26.9977	.0432	43.2	43.1
	2	......	26.9533	5.0013	26.9954	.0421	42.1	
	2	......	26.9592	5.0072	27.0033	.0441	44.0	
3	3	22.0357	27.0351	4.9994	27.1036	.0685	68.5	66.6
	3	......	27.0349	4.9982	27.0999	.0650	65.0	
	3	......	27,0382	5.0025	27.1044	.0662	66.2	
Check	1	24.6902	29.6885	4.9983	29.6935	0.0050	5.0	5.0

TABLE VI

Gas Adsorbed, Carbon Disulphide; Temperature, 35° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6896	4.9997	29.6917	0.0021	2.1	1.8
	2	21.9520	26.9542	5.0022	26.9570	.0028	2.8	
	3	22.0357	27.0458	5.0101	27.0455	— .0003	— .3	
	1	24.6902	29.6923	5.0021	29.6930	.0007	0.7	
	2	21.9520	26.9344	5.0024	26.9561	.0017	1.7	
	3	22.0357	27.0389	5.0032	27.0422	.0033	3.3	
2	1	24.6902	29.6890	4.9988	29.7178	.0288	28.8	29.0
	2	21.9520	26.9596	5.0076	26.9885	.0289	28.8	
	3	22.0357	27.0259	4.9902	27.0554	.0295	29.5	
3	1	24.6902	29.6966	5.0064	29.7558	.0592	59.1	56.7
	2	21.9520	26.9560	5.0040	27.0140	.0580	57.9	
	3	22.0357	27.0365	5.0008	27.0895	.0530	53.0	
4	1	24.6902	29.6879	4.9977	29.7753	.0874	87.4	87.4
5	2	21.9520	26.9516	4.9996	27.0663	.1147	114.7	114.7
Check	3	22.0357	27.0338	4.9981	27.0300	—0.0038	—3.8	—3.8

TABLE VII

Gas Adsorbed, Carbon Disulphide; Temperature, 30° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6865	4.9963	29.6929	0.0064	6.4	5.7
	2	21.9520	26.9611	5.0091	26.9667	.0056	5.6	
	3	22.0357	27.0329	4.9972	27.0381	.0052	5.2	
2	1	24.6902	29.6946	5.0044	29.7271	.0325	32.4	32.2
	2	21.9520	26.9556	5.0036	26.9890	.0334	33.3	
	3	22.0357	27.0391	5.0034	27.0702	.0311	31.1	
3	1	24.6902	29.6851	4.9949	29.7504	.0653	65.3	62.9
	2	21.9520	26.9601	5.0081	27.0226	.0625	62.4	
	3	22.0357	27.0360	5.0003	27.0971	.0611	61.1	
4	1	24.6902	29.6836	4.9934	29.7712	.0876	87.7	87.7
5	2	21.9520	26.9544	5.0024	27.0686	.1142	114.2	114.2
Check	3	22.0357	27.0377	5.0020	27.0338	—0.0039	—3.9	—3.9

TABLE VIII

Gas Adsorbed; Carbon Disulphide; Temperature, 25° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6892	4.9990	29.6981	0.0089	8.9	9.2
	2	21.9520	26.9518	4.9998	26.9606	.0088	8.8	
	3	22.0357	27.0304	4.9947	27.0404	.0100	10.0	
2	1	24.6902	29.6898	4.9996	29.7234	.0336	33.6	31.7
	2	21.9520	26.9572	5.0052	26.9898	.0326	32.5	
	3	22.0357	27.0334	4.9977	27.0624	.0290	29.0	
3	1	24.6902	29.6860	4.9958	29.7530	.0670	67.0	65.7
	2	21.9520	26.9525	5.0005	27.0193	0668	66.8	
	3	22.0357	27.0388	5.0031	27.1022	.0634	63.3	
4	1	24.6902	29.6883	4.9981	29.7763	.0880	88.0	88.0
5	2	21.9520	26.9568	5.0048	27.0820	.1252	125.1	125.1
Check	3	22.0357	27.0332	4.9975	27.0370	0.0038	3.8	3.8

TABLE IX

Gas Adsorbed, Carbon Disulphide; Temperature, 20° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6833	4.9931	29.6961	0.0128	12.8	12.4
	2	21.9520	26.9558	5.0038	26.9677	.0119	11.9	
	3	22.0357	27.0319	4.9962	27.0443	.0124	12.4	
2	1	24.6902	29.6914	5.0012	29.7296	.0382	38.2	38.5
	2	21.9520	26.9511	4.9991	26.9910	.0399	39.9	
	3	22.0357	27.0371	5.0014	27.0745	.0374	37.4	
3	1	24.6902	29.6882	4.9980	29.7574	.0692	69.2	69.7
	2	21.9520	26.9564	5.0042	27.0251	.0687	68.6	
	3	22.0357	27.0384	5.0027	27.1097	.0713	71.3	
4	1	24.6902	29.6907	5.0005	29.7774	.0867	86.7	86.7
5	2	21.9520	26.9556	5.0036	27.0736	.1180	118.0	118.0
Check	3	22.0357	27.0298	4.9941	27.0284	—0.0014	—1.5	—1.5

TABLE X

Gas Adsorbed, Carbon Disulphide; Temperature, 15° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	64.6902	29.6870	4.9968	29.7041	0.0171	17.1	17.1
2	2	21.9520	26.9503	4.9983	26.9912	.0409	40.9	40.9
3	3	22.0357	27.0377	5.0020	27.1084	.0707	70.7	70.7
Check	1	24.6902	29.6914	5.0012	29.6919	0.0005	0.5	0.5

TABLE XI

Gas Adsorbed, Carbon Tetrochloride; Temperature, 35° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6891	4.9989	29.6995	0.0104	10.4	11.8
	2	21.9520	26.9574	5.0054	26.9704	.0130	12.9	
	3	22.0357	27.0322	4.9965	27.0444	.0122	12.2	
2	1	24.6902	29.6937	5.0035	29.7273	.0336	33.6	35.0
	2	21.9520	26.9559	5.0039	26.9924	.0365	36.5	
	3	22.0357	27.0263	4.9906	27.0631	.0348	34.8	
3	1	24.6902	29.6881	4.9979	29.7494	.0613	61.3	61.9
	2	21.9520	26.9598	5.0078	27.0249	.0651	65.0	
	3	22.0357	27.0365	5.0008	27.0960	.0595	59.5	
4	1	24.6902	29.6915	5.0013	29.7860	.0945	94.5	94.5
5	2	21.9520	26.9751	5.0231	27.0829	.1078	107.3	108.3
Check	3	22.0357	27.0396	5.0039	27.0344	—0.0052	—5.2	—5.2

TABLE XII

Gas Adsorbed, Carbon Tetrachloride; Temperature, 30° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6817	4.9915	29.6965	0.0148	14.8	14.9
	2	21.9520	26.9589	5.0069	26.9740	.0151	15.1	
	3	22.0357	27.0245	4.9888	27.0379	.0135	13.5	
	2	21.9520	26.9611	5.0091	26.9786	.0175	17.4	
	3	22.0357	27.0369	5.0012	27.0507	.0138	13.8	
2	1	24.6902	29.6923	5.0021	29.7338	.0415	41.5	42.4
	2	21.9520	26.9664	5.0144	27.0117	.0453	45.2	
	3	22.0357	27.0307	4.9950	27.0713	.0406	40.6	
3	1	24.6902	29.6906	5.0004	29.7623	.0717	71.7	68.0
	2	21.9520	26.9620	5.0100	27.0310	.0690	68.6	
	3	22.0357	27.0304	4.9947	27.0938	.0634	63.4	
4	1	24.6902	29.6847	4.9945	29.7710	.0863	86.4	86.4
5	2	21.9520	26.9567	5.0047	27.0811	.1244	124.3	124.3
Check	3	22.0357	27.0333	4.9976	27.0325	—0.0008	—0.8	—0.8

TABLE XIII

Gas Adsorbed, Carbon Tetrachloride; Temperature, 25° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6885	4.9983	29.7076	0.0191	19.1	19.8
	2	21.9520	26.9524	5.0004	26.9738	.0214	21.4	
	3	22.0357	27.0321	4.9964	27.0508	.0187	18.8	
2	1	24.6902	29.6958	5.0056	29.7437	.0479	46.8	46.3
	2	21.9520	26.9593	5.0073	27.0075	.0462	46.1	
	3	22.0357	27.0317	4.9960	27.0766	.0449	45.0	
3	1	24.6902	29.6914	5.0012	29.7642	.0728	72.6	71.1
	2	21.9520	26.9518	4.9998	27.0218	.0700	70.0	
	3	22.0357	27.0337	4.9980	27.1043	.0706	70.6	
4	1	24.6902	29.6856	4.9954	29.7805	.0949	95.0	95.0
5	2	21.9520	26.9526	5.0006	27.0735	.1209	120.9	120.9
Check	3	22.0357	27.0357	5.0000	27.0329	—0.0028	—2.8	—2.8

TABLE XIV

Gas Adsorbed, Carbon Tetrachloride; Temperature, 20° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6872	4.9970	29.7081	0.0209	20.9	20.2
	2	21.9520	26.9560	5.0040	26.9745	.0185	18.4	
	3	22.0357	27.0332	4.9975	27.0545	.0213	21.3	
2	1	24.6902	29.6910	5.0008	29.7371	.0461	46.1	46.1
	2	21.9520	27.0366	5.0846	27.0822	.0456	45.0	
	3	22.0357	27.0381	5.0024	27.0853	.0472	47.2	
3	1	24.6902	29.6852	4.9950	29.7570	.0718	71.8	71.1
	2	21.9520	26.9520	5.0000	27.0212	.0692	69.2	
	3	22.0357	27.0339	4.9982	27.1062	.0723	72.3	
4	1	24.6902	29.6841	4.9939	29.7740	.0899	90.0	90.0
5	2	21.9520	26.9610	5.0090	27.0709	.1099	109.7	109.7
Check	3	22.0357	27.0359	5.0002	27.0412	.0053	5.3	5.3
	3	......	27.0369	5.0012	27.0423	0.0054	5.4	

TABLE XV

Gas Adsorbed, Carbon Tetrachloride; Temperature, 15° C.; R. H., 50 per Cent; Time, 1 Hour; Concentration per 1000 Cubic Feet

Concentration	Tube No.	Weight of tube and stopper	Weight of tube, stopper, and charcoal	Net weight charcoal	Second weight total	Increase	Increase per 5 gm. charcoal	Average
lb.		gm.	gm.	gm.	gm.	gm.	mg.	mg.
1	1	24.6902	29.6902	5.0000	29.7112	0.0210	21.0	22.0
	1	......	29.6919	5.0017	29.7140	.0221	22.1	
	1	......	28.6897	4.9995	29.7126	.0229	22.9	
2	2	21.9520	26.9550	5.0030	27.0035	.0485	48.5	47.9
	2	......	26.9510	4.9990	26.9983	.0473	47.3	
	2	......	26.9599	5.0079	27.0080	.0481	48.0	
3	3	22.0357	27.0371	5.0014	27.1107	.0736	73.6	72.8
	3	......	27.0392	5.0035	27.1112	.0720	71.9	
	3	......	27.0394	5.0037	27.1125	.0731	73.0	
Check	1	24.6902	29.6952	5.0050	29.7015	0.0063	6.3	6.3